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IS 11657 (1986): Phosphorus oxychloride, technical [CHD 1:
Inorganic Chemicals]

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Indian Standard
SPECIFICATION FOR
PHOSPHORUS OXYCHLORIDE, TECHNICAL

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR

PHOSPHORUS OXYCHLORIDE, TECHNICAL

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Indian Standard

SPECIFICATION FOR

PHOSPHORUS OXYCHLORIDE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 February 1986, after the draft finalized by the Acids, Alkalies and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 Phosphorus oxychloride finds wide use as a catalyst and chlorinating agent in dyestuff industries, pesticides and pharmaceutical industries. This standard, however, does not cover pharmaceutical grade.

0.3 Phosphorus oxychloride is an important starting product for the manufacture of phosphoric acid, triesters, obtained from reactions of POCl_3 with alcohols, phenols and epoxides. These esters are used in plasticizers as additives for motor fuels and lubricating oils.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for phosphorus oxychloride, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a colourless to pale yellow fuming liquid with irritating odour.

*Rules for rounding off numerical values (*revised*).

2.2 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of Table 1.

TABLE 1 REQUIREMENTS FOR PHOSPHORUS OXYCHLORIDE

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Boiling range (95 percent distillation by volume)	104 to 108°C	A-2
ii)	Relative density at 27°C	1.64 to 1.67	A-3
iii)	Assay (as POCl_3), per cent by mass, Min	98	A-4

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in drums with polyethylene liners, in high density polyethylene carboys or in clean glass carboys.

3.2 Marking — The containers shall be marked legibly and indelibly with the following information:

- Name of the material;
- Name of the manufacturer and recognized trade-mark, if any;
- Net mass and batch No.; and
- Date of manufacture.

NOTE — The word 'Poisonous' together with the symbol as given in Fig. 15 of IS : 1260-1973* shall also be given on the label. The label shall have a note as follows:

CAUTION — Phosphorus oxychloride is highly corrosive. When mixed with water, it decomposes with evolution of heat.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

*Pictorial markings for handling and labelling of goods: Part 1 Dangerous goods.

4. SAMPLING

4.1 Representative samples of the material shall be drawn in accordance with the method prescribed in Appendix B.

A P P E N D I X A (Clause 2.2)

METHODS OF TEST FOR PHOSPHORUS OXYCHLORIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. BOILING RANGE

A-2.1 Apparatus

A-2.1.1 *Round Bottom Flask* — of the shape and dimensions as shown in Fig. 1.

A-2.1.2 *Thermometer* — 98 to 152°C.

A-2.1.3 *Leibig Condenser* — Band with long stem as shown in Fig. 2.

A-2.1.4 *Receiver Measuring Cylinder* — 100 ml with the dimensions and graduations as shown in Fig. 3.

A-2.1.5 *Glass Beads*

A-2.2 Procedure — Assemble the apparatus as shown in Fig. 4. Take 100 ml sample in the round bottom flask. Heat it, then note down reading (that is, temperature of very first drop) that is starting temperature. Then take a reading after 5 ml and another after 95 ml keeping a rate of 120 to 125 drops per minute.

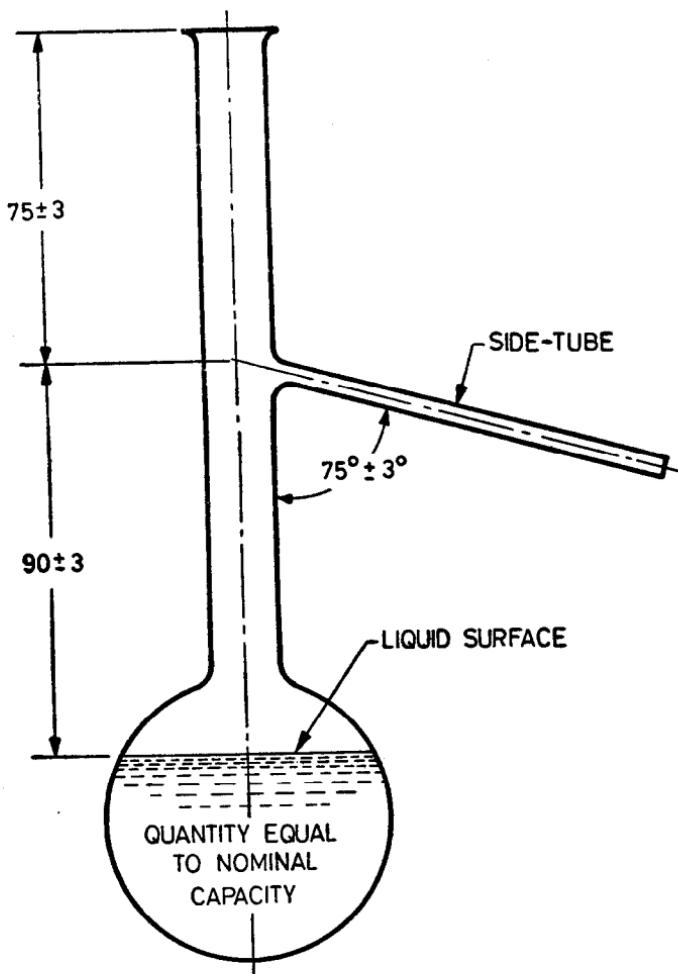
A-2.3 Result — Boiling range = $x^{\circ}\text{C}/5 \text{ ml}$ to $y^{\circ}\text{C}/95 \text{ ml}$.

where

x = temperature at 5 ml, and

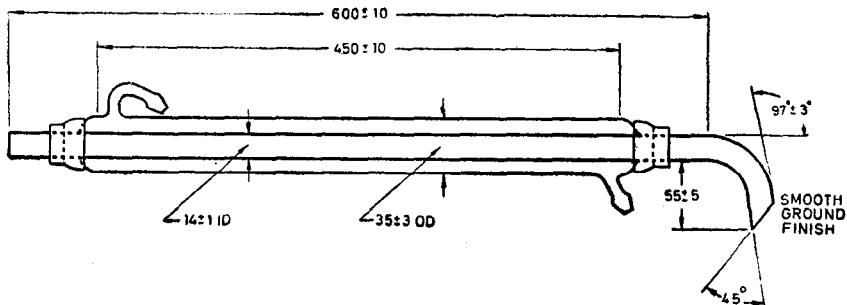
y = temperature at 95 ml.

*Specification for water for general laboratory use (second revision).



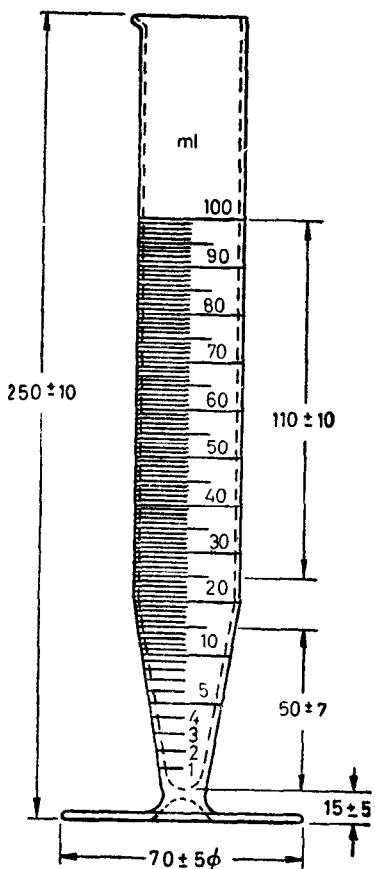
All dimensions in millimetres.

FIG. 1 DISTILLATION FLASK



All dimensions in millimetres.

FIG. 2 LEIBIG CONDENSER



All dimensions in millimetres.

FIG. 3 RECEIVER

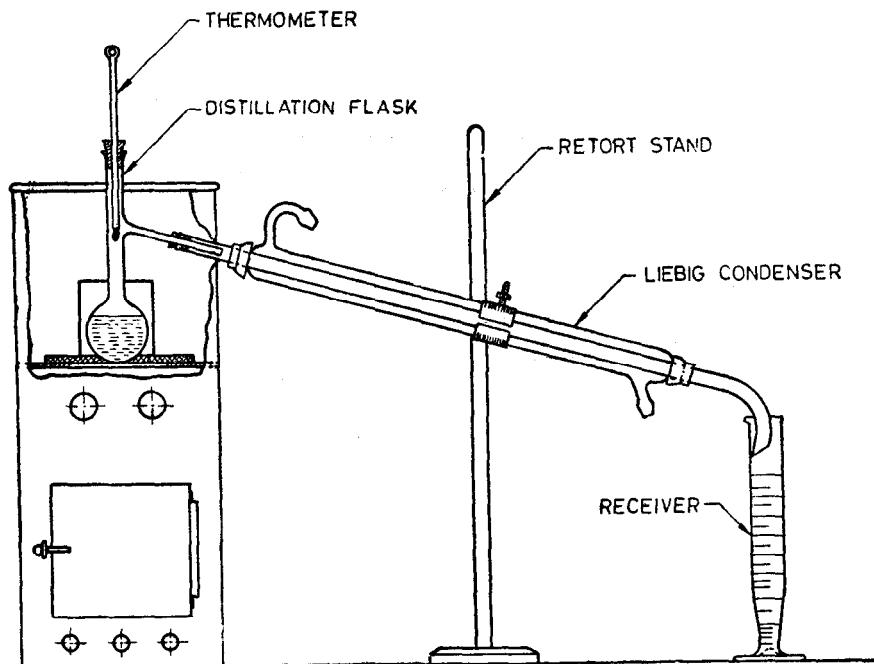


FIG. 4 ASSEMBLY OF APPARATUS

A-3. DETERMINATION OF RELATIVE DENSITY

A-3.1 Apparatus

A-3.1.1 *Hydrometer* — 1 600 to 1 700.

A-3.1.2 *Cylinder* — 250 ml.

A-3.1.3 *Thermometer*

A-3.2 Procedure — Pour the material to be tested into the clean hydrometer jar, the diameter of which shall be at least 2.5 cm greater than the diameter of the hydrometer used. Remove all air bubbles that might have formed in the liquid. Keep the jar in a vertical position and in the bath maintained at 30°C. When the sample in the jar attains the temperature of the bath, that is 30°C, lower the hydrometer gently into the material. When it has settled depress it about two scale divisions into the liquid. Keep the unimmersed portion of the stem dry as any unnecessary liquid on the stem will change the effective weight of the instrument and affect the reading obtained. Allow the hydrometer to

become stationary. Remove all air bubbles that might have formed during lowering of the hydrometer. Read the point on the hydrometer scale to which the sample rises with the eye placed at the principal surface of the material. This reading gives the relative density of the material under test.

A-4. DETERMINATION OF PHOSPHORUS OXYCHLORIDE

A-4.0 Two methods, namely, acidimetric and excess chloride method have been prescribed for this determination. Excess chloride method shall be the referee method.

A-4.1 Assay (Acidimetric Method)

A-4.1.0 Outline of the Method — Phosphorus oxychloride is hydrolyzed by water to give phosphoric acid and hydrochloric acid. This is determined by acid base titration.

A-4.1.1 Reagents

A-4.1.1.1 Standard sodium hydroxide — 0·1 N.

A-4.1.1.2 Thymolphthalein indicator — 0·1 percent.

A-4.1.2 Apparatus

A-4.1.2.1 pH meter

A-4.1.3 Procedure — Weigh accurately about 1 g of the sample (use a stoppered weighing bottle) in about 100 ml of water and about 30 g of sodium chloride. Add about 0·5 ml of thymolphthalein indicator solution and titrate with 0·1 N sodium hydroxide to a blue colour (not green) pH 9·6. For more accurate work, use a pH meter.

A-4.1.4 Calculation

$$\text{Phosphorus oxychloride (POCl}_3\text{), } = \frac{V \times N \times 3\cdot07}{M}$$

percent by mass

where

V = volume in ml of sodium hydroxide,

N = normality of sodium hydroxide, and

M = mass in g of sample taken for test.

A-4.2 Assay (Excess Chloride Method)

A-4.2.0 Outline of the Method — Phosphorus oxychloride is hydrolyzed by water to give phosphoric acid and hydrochloric acid. Hydrochloric acid is determined by excess chloride.

A-4.2.1 Reagents

A-4.2.1.1 Standard silver nitrate solution — 0·1 N.

A-4.2.1.2 Standard ammonium thiocyanate solution — 0·1 N.

A-4.2.1.3 Ferric alum indicator — 40 percent.

A-4.2.1.4 Dilute nitric acid — 6 N.

A-4.2.1.5 Nitrobenzene

A-4.2.2 Procedure — Weigh accurately about 0·2 g of sample in about 50 ml of water contained in a conical flask. Shake well and add 50 ml N/10 silver nitrate and 5 ml of 6 N nitric acid. Add 1 ml ferric alum indicator and 5 ml of nitrobenzene. Back titrate with N/10 ammonium thiocyanate. End point will be brown colour. Carry out a blank filtration.

A-4.2.3 Calculation

$$\text{Phosphorus oxychloride (POCl}_3\text{), } = \frac{(V_1N_1 - V_2N_2) \times 0.51}{M}$$

percent by mass

where

V_1 = volume in ml of silver nitrate,

N_1 = normality of silver nitrate,

V_2 = volume in ml of ammonium thiocyanate,

N_2 = normality of ammonium thiocyanate, and

M = mass in g of sample taken for test.

A P P E N D I X B

(Clause 4.1)

SAMPLING

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, storing, preparing, and handling test samples the precautions given in **B-1.1** to **B-1.5** shall be observed.

B-1.1 Samples shall not be taken at a place exposed to weather.

B-1.2 Precautions shall be taken to protect the samples, the sampling instruments and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.4 The samples shall be placed in suitable, clean dry and air-tight containers.

B-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of phosphorus oxychloride of the same grade drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different batches, the batches shall be marked separately and the groups of containers in each batch constitute a separate lot.

B-2.2 The number of containers (n) to be selected from the lot shall depend upon the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE N	NO. OF CONTAINERS TO BE SELECTED n
Up to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

B-2.3 These containers shall be selected at random from the lot and in order to ensure the randomness of selection, procedures given in IS : 4905-1968* may be followed.

B-2.4 Samples shall be tested for each lot for ascertaining conformity of the material to the requirements of the specification.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected according to **B-2.3** a portion of the liquid, about 200 ml, shall be drawn with the help of a suitable sampling instrument.

*Methods for random sampling.

B-3.2 Out of these portions drawn from each container, equal quantities of liquid shall be taken and mixed thoroughly to form a composite sample of 600 ml. The composite sample shall be divided into three parts, one for the purchaser, another for the supplier and the third used as a referee sample.

B-3.3 The remaining portion of the liquid drawn from each container, shall be divided into three equal parts each forming an individual sample. One set of individual sample representing 'N' containers sampled shall be marked for the purchaser, another for the supplier and the third used as a referee sample.

B-3.4 All the individual samples and the composite samples shall be transferred to separate sample containers. All the containers shall be sealed and labelled with full identification particulars.

B-3.5 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier to be used in case of a dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of phosphorus oxychloride content shall be performed on each of the individual samples.

B-4.2 Tests for the determination of all other characteristics given in Table I shall be performed on the composite sample.

(Continued from page 2)

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²